Geophysical Research Abstracts
Vol. 18, EGU2016-15643, 2016
EGU General Assembly 2016

The next chapter in experimental petrology: Metamorphic dehydration of polycrystalline gypsum captured in 3D microtomographic time series datasets

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Nucleation and growth of new minerals in response to disequilibrium is the most fundamental metamorphic process. However, our current kinetic models of metamorphic reactions are largely based on inference from fossil mineral assemblages, rather than from direct observation. The experimental investigation of metamorphism has also been limited, typically to concealed vessels that restrict the possibility of direct microstructural monitoring. Here we present one of the first time series datasets that captures a metamorphic reaction, dehydration of polycrystalline gypsum to form hemihydrate, in a series of three dimensional x-ray microtomographic datasets.

We achieved this by installing an x-ray transparent hydrothermal cell (Fusseis et al., 2014, J. Synchrotron Rad. 21, 251-253) in the microtomography beamline 2BM at the Advanced Photon Source (USA). In the cell, we heated a millimetre-sized sample of Volterra Alabaster to 388 K while applying an effective pressure of 5 MPa. Using hard x-rays that penetrate the pressure vessel, we imaged the specimen 40 times while it reacted for approximately 10 hours. Each microtomographic dataset was acquired in 300 seconds without interrupting the reaction. Our absorption microtomographic data have a voxel size of 1.3 μm, which suffices to analyse the reaction progress in 4D.

Gypsum can clearly be distinguished from hemihydrate and pores, which form due to the large negative solid volume change. On the resolved scale, the first hemihydrate needles appear after about 2 hours. Our data allow tracking of individual needles throughout the entire experiment. We quantified their growth rates by measuring their circumference. While individual grains grow at different rates, they all start slowly during the initial nucleation stage, then accelerate and grow steadily between about 200 and 400 minutes before reaction rate decelerates again.

Hemihydrate needles are surrounded by porous haloes, which grow with the needles, link up and eventually encapsulate the remaining gypsum crystals. The reaction appears to be homogenously distributed throughout the sample and we find no evidence for metamorphic overpressure. We used an advanced machine learning algorithm (http://fiji.sc/Trainable_Weka_Segmentation) to segment the porosity from the microtomographic data and quantify it in Fiji (Schindelin et al., 2012, Nature Methods 9, 676–682). The porosity evolution follows the grain growth curves and reaches 23%, which indicates that the dehydration reaction is 80% complete.

Our 4D data provide a unique opportunity not only to explore evolving reaction microtextures in spectacular visualisations but also to test general metamorphic theory. Our data, which track the entire reaction history from nucleation through to interaction with surrounding grains raise several questions. While individual grains grow quicker than others, why do, when grain growth is normalised against final grain size, all grains have a very similar growth history? This is not currently explained by an Avrami type model, and an alternative model for metamorphic reaction kinetics may be based on our data. Do metamorphic transport distances change during the reaction as the pore structure evolves? What controls the orientation of hemihydrate needles? In this presentation we present not only the images and data highlighting these questions, but also explore possible answers.